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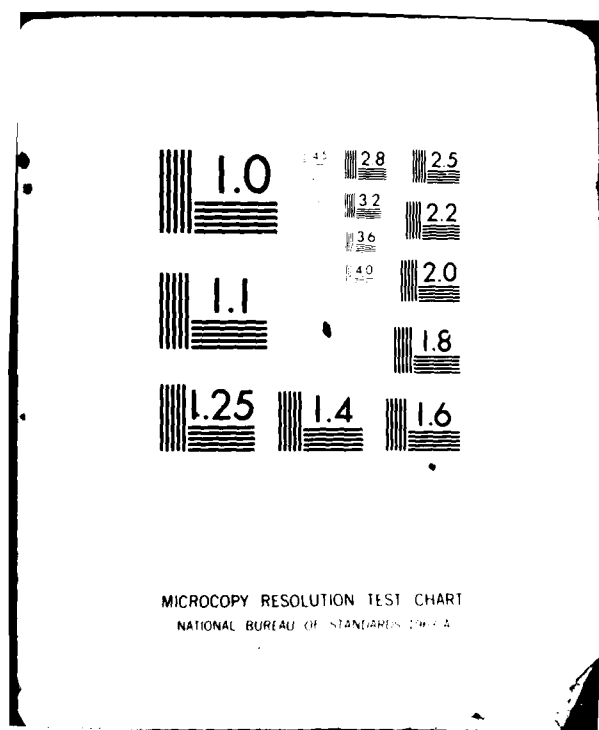
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3rd SYMPOSIUM ON APPLIED SURFACE ANALYSIS

.by

JOHN T. GRANT

Final Technical Report

March 1982

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UNIVERSITY OF DAYTON
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20. ABSTRACT (continued)

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FOREWORD

This report describes the 3rd Symposium on Applied Surface Analysis held at the University of Dayton, 300 College Park Avenue, Dayton, Ohio 45469, on 3-5 June 1981, under AFOSR-~~81-0159~~ 81-0159.

The Symposium was conducted by the University of Dayton with Dr. John T. Grant of that organization and Dr. T. W. Haas of the Materials Laboratory, Air Force Wright Aeronautical Laboratories as Co-Chairmen. This report was submitted in March 1982.

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SECTION I

INTRODUCTION

The 3rd Symposium on Applied Surface Analysis was held at the University of Dayton, 3-5 June 1981. This Symposium was held to meet a need, namely to show the transition between basic surface science research and applications of this research to areas of Air Force interest. Areas receiving special attention at this Symposium were adhesion, coatings, corrosion, catalysis, thin films, and electronic materials. Other topics discussed included adsorption, wear and developments in secondary ion mass spectroscopy. Approximately 130 scientists active in the field of surface analysis participated in the Symposium. Five scientists presented invited papers at the Symposium. There were 45 contributed presentations. The proceedings of the Symposium are being published in a special issue of the journal "Applications of Surface Science" by North-Holland Publishing Company.

SECTION II

SYMPOSIUM GOALS

The 3rd Symposium on Applied Surface Analysis was held at the University of Dayton, 3-5 June 1981. This Symposium was organized to meet a need, namely, to show the continuing transition between basic research and applications of this research to areas of Air Force interest. Workers engaged in basic research and those engaged in applications of surface science research were brought together at the Symposium in an environment suitable for promoting the maximum possible interaction between such workers.

The Symposium was planned and organized by the University of Dayton, with Dr. John T. Grant of the University of Dayton and Dr. T. W. Haas of the Materials Laboratory, Air Force Wright Aeronautical Laboratories as Co-Chairmen. Five invited speakers were selected to cover specific areas of interest. These speakers and their topics were:

1. J. T. Yates
National Bureau of Standards
"Spectroscopic Correlation in Surface Chemistry -
Forging a Relationship Between Chemisorption on
Supported Metals, on Single Crystals, and To Bonding
in Organometallic Chemistry"
2. K. Wittmaack
City of London Polytechnic
"Recent Advances in Secondary Ion Mass Spectroscopy"
3. C. A. Evans, Jr.
Evans & Associates
"High Performance SIMS for Electronic Material
Analysis"
4. W. E. Spicer
Stanford University
"A Unified Model for Formation of Schottky Barrier
and Oxide-Semiconductor States on 3-5 Compounds and
Implications Theory"
5. D. H. Buckley
NASA-Lewis Research Center
"The Use of Analytical Surface Tools in the Fundamental
Study of Adhesion, Friction and Wear"

Contributed papers on applied surface analysis were also solicited from the research community, resulting in 45 such papers being presented at the Symposium.

It was decided to publish as many of the papers as possible in an appropriate journal, thereby providing a permanent record of the work presented at the Symposium. The journal "Applications of Surface Science" was selected, and the journal editor was pleased to be able to publish such a proceedings. Thirty manuscripts were submitted for peer review and eventual publication in the proceedings. It is hoped that the proceedings will be published and distributed to all attendees in the Spring of 1982.

The Symposium was advertised through appropriate scientific societies, equipment manufacturers, scientific journals, and at other meetings. It was decided to hold the Symposium on the University of Dayton campus and to encourage attendees to stay in University apartments in order to provide an informal atmosphere and to optimize the interaction between attendees.

Two events were organized by the University and held in conjunction with the Symposium, namely a two-day short course and a vendors' exhibit. The short course topic was "Techniques for Surface Analysis," and was held immediately before the Symposium to provide an opportunity for attendees to learn the fundamentals of several surface analysis techniques, so that they could gain as much as possible from the Symposium presentations and in their own research. The vendors' exhibit provided an opportunity for attendees to learn about the commercial surface instrumentation that is currently available and what is planned for the future.

SECTION III
PARTICIPANTS

Approximately 130 scientists active in the field of surface analysis attended the Symposium.

The list of participants follows.

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SECTION IV PROGRAM

The technical program was divided into six sessions. Five of the sessions started with a presentation by one of the invited speakers.

The program organization and the abstracts of the papers presented at the Symposium follow.

3RD SYMPOSIUM ON
APPLIED SURFACE ANALYSIS

FINAL PROGRAM

Supported by

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

3 - 5 JUNE 1981
UNIVERSITY OF DAYTON
DAYTON, OHIO

3RD SYMPOSIUM ON APPLIED SURFACE ANALYSIS
3-5 JUNE 1981
UNIVERSITY OF DAYTON

General Information

The 3rd Symposium on Applied Surface Analysis is being supported by the Air Force Office of Scientific Research. The Symposium will provide an opportunity for researchers in all areas of surface characterization to meet and discuss applications of surface analysis. It is the interest of the Symposium Committee to attract participants from universities, and from government, nonprofit and industrial laboratories to promote interaction and technical exchange among participants.

All sessions will be held in O'Leary Auditorium located in Miriam Hall on the University of Dayton campus.

The Symposium headquarters will be adjacent to the O'Leary Auditorium located in Miriam Hall on the University of Dayton campus. Emergency telephone messages can be taken at (513) 229-2113 during the Symposium. Several telephones are available for use in Kennedy Union.

Attendees staying in University apartments can pick up their keys from Patrick O'Rourke in Apartment 2A, 363 Stewart Street, upon arrival. Check out time is 12 Noon.

Limousine service is available between downtown Dayton and Dayton International Airport. Telephone (513) 898-1012 for schedule and costs. Taxi service from campus to downtown is approximately \$5.00. City bus service (#5 Far Hills and #12 Belmont, southbound) leaves downtown Third and Main Streets and stops within a few blocks of the apartments.

Symposium registration will be held during the Symposium, starting at 8:00 a.m. on Wednesday, 3 June. All attendees must be registered.

Proceedings

The proceedings will be published in a special issue of "Applications of Surface Science." Manuscripts (original plus three copies) should be delivered to the Symposium Secretary at the Registration Desk.

Vendors Exhibits & Reception

A vendor's exhibit will be held from noon Wednesday, 3 June, through 3:30 p.m. Thursday, 4 June. Refreshments will be served in the vendor's area at the conclusion of the afternoon session on Wednesday. An informal reception for all attendees will be held in Kennedy Union at the conclusion of the afternoon session on Thursday.

Physical Activities Center

The Physical Activities Center on the University of Dayton campus will be available for use by the conference attendees at a cost of \$1.00 per visit. Various activities at the facility include swimming, jogging, basketball, racquet ball, and handball.

SYMPOSIUM ADMINISTRATION

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AGENDA

WEDNESDAY MORNING, 3 JUNE 1981

SESSION A

Chairman: Dr. C. G. Pantano
Pennsylvania State University

- 8:00 REGISTRATION IN MIRIAM HALL
- 9:00 INTRODUCTORY REMARKS
- A-1 9:20 J. T. Yates (Invited), "Spectroscopic Correlation in Surface Chemistry - Forging a Relationship Between Chemisorption on Supported Metals, on Single Crystals, and To Bonding in Organometallic Chemistry"
- A-2 10:10 J. S. Jen, "ESCA Study of Gama-Aminopropyltriethoxysilane Adsorbed on Silica and Sodium Silicate Glass Surfaces"
- 10:30 COFFEE BREAK
- A-3 10:50 C. R. Anderson, S. D. James, W. R. Kilroy, and R. N. Lee, "Surface Analysis of Li/SO₂ Battery Electrodes"
- A-4 11:10 P. A. Lindfors, "Computer Curve Fitting of ESCA Data to Aid Interpretation"
- A-5 11:30 L. Ranta and E. Suoninen, "Studies of Surface Processes in Flotation of Sulfide Minerals"
- A-6 11:50 W. V. Lambert, W. L. Baun, B. C. Lamartine, and T. W. Haas, "XPS/ISS Investigation of the Activation of M-Type Impregnated Dispenser Cathodes"
- A-7 12:10 R. C. McCune and P. Wynblatt, "The Use of AES and ISS Techniques for Measurement of the Segregation of Calcium to the MgO (100) Surface"
- 12:30 LUNCH
- 12:00 VENDORS' EXHIBITS OPEN
- Noon

WEDNESDAY AFTERNOON, 3 JUNE 1981

SESSION B

Chairman: J. S. Solomon
University of Dayton

- B-1 1:30 K. Wittmaack (Invited), "Recent Advances in Secondary Ion Mass Spectrometry"
- B-2 2:20 M. Schemmer, P. Beckmann, D. Greifendorf, and A. Benninghoven, "Secondary Ion Emission From Adsorbate Layers on Polycrystalline Ni"
- B-3 2:40 G. Smith, C. J. Brinker, and C. G. Pantano, "Secondary Ion Mass Spectroscopy Studies of Glass Thin Films Prepared with Sol-Gel Techniques"
- 3:00 COFFEE BREAK
- B-4 3:30 S. Matteson, "Mass Redistribution by Atomic Mixing in Sputter Depth Profiling"
- B-5 3:50 S. C. Tjong, "ESCA and SIMS Analysis of the Composition of Passive Films Formed on E-Brite Stainless Steel"
- B-6 4:10 D. F. Mitchell, "Quantitative Interpretation of Auger Sputter Profiles of Thin Layers"
- B-7 4:30 H. Yamada and E. L. Wood, "Determination of Subsurface Helium Distribution by Ar⁺ Sputtering and Residual Gas Analysis (RGA)"
- B-8 4:50 D. J. Hunt, D. T. Peters, J. W. Wilson, and H. L. Yeh, "Applications of AES to Failure Analysis in Commercial Nickel and Copper Alloys"
- 5:10 REFRESHMENTS
- 6:00 VENDORS' EXHIBITS CLOSED FOR TODAY - REOPEN 10:20 a.m. THURSDAY

THURSDAY MORNING, 4 JUNE 1981

SESSION C

Chairman: Dr. Y. S. Park
Avionics Laboratory

- C-1 8:30 C. A. Evans, Jr. (Invited), "High Performance SIMS for Electronic Material Analysis"
- C-2 9:20 B. F. Phillips, "Quadrupole Mass Spectrometer Based Secondary Ion Mass Spectrometry for Electronic Materials Characterization"
- C-3 9:40 W. Katz, O. Aina, B. J. Baliga, and K. Rose, "SIMS Analysis of Low Temperature Ohmic Contacts to GaAs"
- C-4 10:00 K. Y. Chang and R. K. Panchohy, "Tantalum Silicide Interconnect Characterization by Surface Analytical Techniques"
- 10:20 COFFEE BREAK
- 10:20 VENDORS' EXHIBITS OPEN
- C-5 10:50 M. B. Chamberlain, "TiN-Cu Metallization for Solar Concentrator Cells"
- C-6 11:10 J. S. Hammond, T. E. Brady, and C. T. Hovland, "Spatially Resolved Chemical Analysis by Auger Spectroscopy"
- C-7 11:30 C. D. Wagner, H. A. Six, and W. T. Jansen, "Improving the Accuracy of Determination of Line Energies by ESCA: Chemical State Plots for Silicon-Aluminum Compounds"
- C-8 11:50 D. J. Surman and J. C. Vickerman, "Surface Analysis by Fast Atom Bombardment Mass Spectrometry"
- 12:10 LUNCH

THURSDAY AFTERNOON, 4 JUNE 1981

SESSION D

Chairman: Dr. J. Comas
Naval Research Laboratory

- D-1 1:30 W. E. Spicer (Invited), I. Lindau, C. Y. Su, and P. Skeath, "A Unified Model for Formation of Schottky Barrier and Oxide-Semiconductor States on 3-5 Compounds and Implications Theory"
- D-2 2:20 G. Le Lay, A. Chauvet, M. Manneville, and R. Kern, "The Structural and Electronic Properties of Cleaved Silicon (111) Surfaces Following Adsorption of Silver"
- D-3 2:40 G. Rossi, I. Abbati, L. Braicovich, I. Lindau, and W. E. Spicer, "Valence Band Synchrotron Radiation Photoemission for Ge (111) /"d" Metal Interfaces: The Ge/Pd, Ge/Ni, Ge/Ag Cases"
- 3:00 COFFEE BREAK
- 3:30 VENDORS' EXHIBITS CLOSED
- D-4 3:30 P. P. Yaney, W. E. Baird, Jr., and Y. S. Park, "Studies of Implanted GaAs Using Raman Scattering"
- D-5 3:50 L. L. Abels, S. Sundaram, R. L. Schmidt, and J. Comas, "Effects of Implantation and Annealing on the Raman Spectrum of InP and GaAs"
- D-6 4:10 I. Golecki, H. L. Glass, G. Kinoshita, and T. J. Magee, "Measurements of Defects and Strain in SOS Films After CW Ar Laser Annealing in the Liquid Phase Regime"
- D-7 4:30 W. L. Baun, "Ubiquitous Sodium: Where Does It Come From?"
- D-8 4:50 N. C. Fernelius, "Surface-to-Bulk Optical Absorption Measurements of Laser Windows Using Photoacoustic Chopping Frequency Studies"
- D-9 5:10 R. G. Wilson, "On the Nature of the Redistribution of Be in Annealed GaAs"
- 5:30 CLOSE
- 5:30 RECEPTION (to 7:00 p.m.)

FRIDAY MORNING, 5 JUNE 1981

SESSION E

Chairman: N. T. McDevitt
Materials Laboratory

- E-1 8:30 D. H. Buckley (Invited), "The Use of Analytical Surface Tools in the Fundamental Study of Adhesion, Friction and Wear"
- E-2 9:20 E. G. Shafrin, "Auger Compositional Analysis of Gear Steel Reacted with Tricresyl Phosphate"
- E-3 9:40 L. J. Matienzo, K. J. Holub, J. J. Mills, "Surface Studies of Corrosion Preventing Coatings for Aluminum Alloys"
- E-4 10:00 G. W. Stupian and P. D. Fleischauer, "Corrosion Protection of Aluminum by Solution-Deposited Mixed Oxide Films"
- 10:20 COFFEE BREAK
- E-5 10:50 E. A. Podoba, S. P. Kodali, and R. C. Curley, "Adhesive Bond Durability with Conversion Coatings"
- E-6 11:10 A. Roche, M. Charbonnier, F. Gaillard, R. Bador, and M. Romand, "Applications of Soft X-Rays in Surface Analysis"
- E-7 11:30 T. S. Sun, G. D. Davis, J. S. Ahearn, and J. D. Venables, "The Hydration of Phosphoric Acid Anodized Aluminum Surfaces: A Surface Phase Diagram Determined by XPS"
- E-8 11:50 K. J. Holub and L. J. Matienzo, "Thermal Diffusion of Magnesium in Several Aluminum Alloys"
- E-9 12:10 I. Krainsky, W. L. Gordon, and R. W. Hoffman, "Electron Beam Induced Damage in ITO Coated Kapton"
- 12:30 LUNCH

FRIDAY AFTERNOON, 5 JUNE 1981

SESSION F

Chairman: Dr. B. C. Lamartine
Materials Laboratory

- | | | |
|-----|------|--|
| F-1 | 1:30 | <u>J. L. Lauer</u> and L. E. Keller, "Analysis of Aircraft Fuel Line Deposits by Polarization Infrared Fourier Micro-Emission Spectrophotometry" |
| F-2 | 1:50 | D. Chadwick and <u>M. A. Karolewski</u> , "Oxidation of Ultra-Thin Lead Films on Polycrystalline Silver Surfaces" |
| F-3 | 2:10 | <u>C. W. Miller</u> and T. Kuwana, "A Versatile Sample Isolation, Chemical Modification and Introduction System Designed for a Physical Electronics Model 548 Electron Spectrometer" |
| F-4 | 2:30 | <u>S. E. Hebboul</u> , R. W. Hoffman, and F. Jansen, "Surface Viscous Flattening Kinetics of Amorphous Selenium by Optical Scattering Technique" |
| F-5 | 2:50 | P. Hudson, J. F. Asmus, and <u>J. H. Brannon</u> , "Citric Acid Augmented Flashlamp Cleaning of Corroded Steel Surfaces" |
| F-6 | 3:10 | J. A. Hain, <u>G. W. Johnson</u> , and J. A. Buono, "The Use of Backscatter Electron Imaging in Inspection and Evaluation of Thick Film Circuitry" |
| F-7 | 3:30 | <u>R. B. Zipin</u> , "The Height Sensitive Surface Roughness Parameters" |
| F-8 | 3:50 | <u>D. W. Savage</u> , "A Study of 946A Connector Thermal-Compression Bond Failure as Related to Surface Texture" |
| F-9 | 4:10 | <u>R. D. Seals</u> , J. S. Bullock, R. K. Bennett, "Residual Contamination and Corrosion on Electrochemically Marked Uranium" |
| | 4:30 | ADJOURNMENT |

SPECTROSCOPIC CORRELATION IN SURFACE CHEMISTRY - FORGING A
RELATIONSHIP BETWEEN CHEMISORPTION ON SUPPORTED METALS, ON SINGLE
CRYSTALS, AND TO BONDING IN ORGANOMETALLIC CHEMISTRY

J. T. Yates, Jr.
Surface Science Division
National Bureau of Standards

In this lecture it will be shown that various methods of studying the vibrational spectrum of an adsorbed species have allowed us to see relationships between diverse areas of surface chemistry. The talk will begin with a summary of results for carbon monoxide chemisorbed on supported Rh as studied by transmission infrared spectroscopy. Comparisons will be made with the vibrational spectrum of CO on a Rh(111) single crystal. These results will be compared with the chemisorptive behavior of electronic analogs to CO and the concept of "anchor group" molecular stabilization at surfaces will be illustrated.

The chemisorption of CO on Pd(210) will be discussed in some detail, and it will be shown that modern methods can actually locate the exact CO bonding site on this surface and that both vibrational spectra and vibrational amplitudes may be directly observed for this species.

Wednesday, 9:20 a.m.

A-1

23

ESCA STUDY OF GAMA-AMINOPROPYLTRIETHOXSILANE
ADSORBED ON SILICA AND SODIUM SILICATE GLASS SURFACES

J. S. Jen

Research and Development Division
Owens-Corning Fiberglas Corporation
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Adsorption of Gama-Amino propyltriethoxysilane (A1100) on silica and sodium silicate (25% mole of Na₂O) glass surfaces was studied by means of ESCA (Electron Spectroscopy for Chemical Analysis). It was found that the substrate surface has a profound influence on the interfacial bonding. For the case of silane adsorption on silica surface, approximately 23% of amino groups of A1100 coupling agent were protonated. Whereas, no protonation was found when sodium silicate glass was used as the substrate. Based on these findings, models are proposed to describe the molecular bonding at the silane/glass interface.

Wednesday, 10:10 a.m.

A-2

24

SURFACE ANALYSIS OF Li/SO₂ BATTERY ELECTRODES

C. R. Anderson, S. D. James, W. R. Kilroy and R. N. Lee

Naval Surface Weapons Center
White Oak, Silver Spring, MD 20910

Both the lithium anode and the high surface-area Shawinigan Black current collector of the Li-LiAsF₆-AN-SO₂ battery system have been analyzed by XPS, SAM and SEM at several stages of battery discharge. The lithium electrode reaction layer, which sometimes explodes, is formed by complex reactions with LiAsF₆, AN and SO₂. The carbon and fluoride surface reaction products are very sensitive to the state of discharge. However, the sulfur reaction products are remarkably stable against discharge. The carbon current collector is known to catalyze violent reactions in the presence of highly dispersed lithium -- a condition which occurs during under-potential discharge. The surface studies of lithium on carbon showed lithium to be much more mobile in -- and more reactive with -- ground graphite than with Shawinigan Black.

Wednesday, 10:50 a.m.

A-3
25

Computer Curve Fitting of ESCA Data
to Aid Interpretation

by

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Physical Electronics Division
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A difficult problem in surface analysis can be to distinguish a thin film coating of one organic material from a substrate of another organic material. If there are differences in the relative concentrations of bonding states of C between the coating material and the substrate material, then computer curve fitting can be used to differentiate the coating from the fiber. Data will be shown from fluorocarbon coatings on polyester fibers and other examples.

In cases where shifts in photoelectron binding energies are not sufficient to distinguish between chemical states and Auger parameter data is not available, then computer curve fitting combined with quantitative calculations can be utilized to identify the compound(s) present. Data will be shown for differentiation between Li_2O , LiOH and Li_2CO_3 . Conclusions reached in this fashion will be compared with identification provided by the shape of the oxygen Auger peak.

Wednesday, 11:10 a.m.

A-4

26

XPS/ISS Investigation of the Activation
of M-Type Impregnated Dispenser Cathodes

W. V. Lampert
W. L. Baun
B. C. Lamartine
and
T. W. Haas

Air Force Wright Aeronautical Laboratories (AFWAL/MLBM)
Wright-Patterson AFB, Ohio 45433

The work function of impregnated tungsten dispenser cathodes can be lowered by several tenths of an electron volt by sputter coating the emitting surface with an 80% Os, 20% Ru coating approximately one micron thick. This result would be expected if the thin film model of dispenser cathode activation is correct. The Os/Ru coating would have a higher work function than bare W, giving rise to a stronger dipole moment when the surface is activated by barium adsorption. This stronger dipole moment results in a lower net work function of the cathode. In this work we report results of an XPS/ISS investigation of the chemical changes on the surfaces of two M-type cathodes: a 4:1:1 and a 5:3:2 BaO:CaO:Al₂O₃ mole impregnant ratio. The Os and Ru XPS peaks show relatively little change in energy during activation, indicating small or no chemical state changes. The XPS Ba peaks, on the other hand, did show changes indicating the adsorption of metallic Ba on the surface. These changes were most pronounced on the X-ray excited Ba MNN Auger peaks and indicated a more metallic Ba layer than was observed on the uncoated impregnated dispenser cathodes. Similar changes are also seen in the ISS data. These changes are probably results of the higher binding energy, hence lower evaporation rate, of metallic Ba from a higher work function substrate.

Wednesday, 11:50 a.m.

A-6
27

THE USE OF AES AND ISS TECHNIQUES FOR MEASUREMENT OF THE SEGREGATION
OF CALCIUM TO THE MgO (100) SURFACE

R. C. McCune and P. Wynblatt
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ABSTRACT

The segregation of impurities to the surfaces of ionic crystals may occur at temperatures of interest in ceramic processing where the free energy of the crystal is reduced by such segregation. This work reports on measurements of the segregation of calcium to the (100) cleavage surface of MgO, using Auger electron spectroscopy (AES) and ion scattering spectroscopy (ISS) under in-situ conditions. In these experiments, the crystal is held at the temperature of interest until the surface composition approaches equilibrium. From values of the surface concentration ratio, X_{Ca}/X_{Mg} , at various temperatures ranging from 900°C to 1450°C, it is possible to determine an experimental heat of segregation, ΔH_{seg} . Values of ΔH_{seg} obtained from ISS and AES are in the range of 11-14 kcal/mole, which is in agreement with an estimate of 14 kcal/mole, arrived at by consideration of lattice strain for Ca in MgO, and reported differences in surface free energy of CaO and MgO. While ΔH_{seg} determined by ISS and AES is in relatively close agreement, ISS indicates an overall greater degree of segregation by roughly a factor of two, using molar concentrations derived from bulk standards. The disruptive effect of both measuring techniques is examined with regard to the practical aspects of determining surface segregation in ceramics.

Wednesday, 12:10 p.m.

A-7

28

'SECONDARY ION MASS SPECTROSCOPY STUDIES OF GLASS THIN FILMS
PREPARED WITH SOL-GEL TECHNIQUES'

G. Smith,[†] C. J. Brinker,* and C. G. Pantano[†]

In the sol-gel process, glass-like macromolecules are formed in solution at low temperature by chemical polymerization. The macromolecules may contain non-network modifying ions as well as various network forming cations linked by bridging oxygens. Polymerization proceeds until the solution is transformed into a stiff, amorphous mass referred to as a 'gel'. Alternatively, the solution may be applied to a metal or ceramic substrate where it polymerizes to form a 'gel film'. The porous gels - typically 100-500 m²/gm - are consolidated by heating at temperatures near the glass transition.

Secondary Ion Mass Spectroscopy (SIMS) has been used to depth profile multicomponent glass films on metal and ceramic substrates in both the gel and densified forms. The gels were densified in oxidizing, reducing, and neutral atmospheres. The interpretation of the SIMS data is shown to have depended significantly upon the structure of the film. Semiquantitative analysis was performed by depositing films upon glass substrates of known composition.

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* Sandia Laboratories, Albuquerque, NM 87115

This work is supported by DOE Contract No. 40-0451.

MASS REDISTRIBUTION BY ATOMIC MIXING
IN SPUTTER DEPTH PROFILING

by: S. Matteson

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A fundamental limitation to the depth resolution of sputter depth profiling in near surface analysis is ion-impact-induced atomic mixing. The quantitative predictions of a recent theoretical model¹ of atomic mixing during ion impact are compared with experimental in-depth secondary ion profiles of originally abrupt mass distributions. A numerical method is presented which accurately accounts for the redistribution of material under conditions of a depth dependent "diffusion-like" transport and surface erosion.

¹ S. Matteson, Applied Physics Letters (in press).

S. Matteson, B.M. Paine, M. -A. Nicolet, Nucl. Inst. Methods (in press).

ESCA and SIMS Analysis of the Composition of Passive Films
Formed on E-Brite Stainless Steel

By S. C. Tjong

Case Laboratories for Electrochemical Studies
Chemistry Department, Case Western Reserve University
Cleveland, Ohio 44106

ABSTRACT

Anodic Oxide Films were formed on E-Brite 26% Cr - 1% Mo stainless steel in 1.0M NaCl + H₂SO₄ solution (pH 3.3). ESCA and SIMS techniques have been used to determine the composition of the passive film. It was found by both techniques that molybdenum was present on the outermost part of the film, however, it was not detected by Auger Electron Spectroscopy as reported by other workers. The results also showed that there was a chromium enrichment and a depletion of iron in the passive film.

Wednesday, 3:50 p.m.

B-5
31

Quantitative Interpretation of Auger Sputter
Profiles of Thin Layers

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ABSTRACT

This paper describes a model which quantitatively evaluates the Auger sputter profiles of thin surface layers, whose thickness is similar to the elastic mean free path of the Auger electrons. In the model, the intensity of an Auger signal is taken as the sum of the signals from each atomic layer, the signal from any one layer being the product of a relative sensitivity factor, an attenuation coefficient related to the mean free path of the particular Auger electron, and the elemental concentration in the layer. The statistical nature of the sputtering process is also incorporated in the form of a modified Poisson distribution. Oxide disproportionation and ion beam mixing have not been considered as they have been minimized experimentally by sputtering with 500ev xenon.

Comparisons between the experimental data and predictions from the model are shown for a number of surface layers, e.g., the "passive" oxide films on Fe and Ni, the electropolish film on Fe-25Cr, and an Fe film electrodeposited onto Ni.

Wednesday, 4:10 p.m.

B-6

32

Determination of Subsurface Helium Distribution by Ar^+ Sputtering
and Residual Gas Analysis (RGA)*

by

H. Yamada

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and

E. L. Wood

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ABSTRACT

An experimental technique has been developed to determine helium distribution profiles in structural materials irradiated in the cores of fast breeder reactors as well as in the blanket region of proposed thermonuclear fusion reactors. The technique utilizes a quadrupole mass spectrometer as residual gas analyzer for measuring the amount of helium released from a specimen surface into a vacuum chamber by Ar^+ sputtering.

Preliminary results from Type 316 stainless steel specimens irradiated in the core of the Experimental Breeder Reactor-II (EBR-II) indicate that the specimens have very high helium concentrations just beneath the surface, which decrease very sharply with increasing depth. The helium distribution profiles observed are characteristic of so-called neutron-induced helium implantation, which occurs in the cores of fast breeder reactors as a result of the elastic scattering of fast neutrons by helium atoms.

Since the observed helium distribution profiles are very different from those produced by bombardment with monoenergetic helium particles, it is not clear whether or not some type of blister will be formed. Surface observations made by SEM, STEM, and Auger spectroscopy to explore the possible presence of such blisters will be discussed.

*Work supported by the U.S. Department of Energy.

Wednesday, 4:30 p.m.

B-7

33

INCO RESEARCH & DEVELOPMENT CENTER, INC.
A UNIT OF INCO CORPORATE R & D
STERLING FOREST
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APPLICATION OF AES TO FAILURE ANALYSIS
IN COMMERCIAL NICKEL AND COPPER ALLOYS

- by -

D. J. Hunt*, D. T. Peters*, J. W. Wilson*, H. L. Yeh**

ABSTRACT

Cracking, tearing, and embrittlement of metals and metal alloys is often a result of segregation of impurity elements to a particular site such as a grain boundary. In many cases, the analytical procedures or instruments available do not provide sufficient sensitivity or resolution to allow observation of the segregated species.

In this paper, a number of very practical examples of failure of commercial materials has been studied by Auger Electron Spectroscopy, and the segregating element or elements identified. Fracture surfaces generated in situ in the high vacuum instrument have been examined in most cases, but we show at least one example where useful information of segregation to grain boundaries was obtained on a specimen fractured at high temperature in air and shipped hundreds of miles to the AES instrument. The Auger analyses are supplemented by bulk chemical analysis and by SEM observation of the fracture surfaces.

The examples described were evidenced by lack of hot ductility of 80%Ni-20%Cr, 70%Cu-30%Ni and 60%Ni-40%Cu alloys, and embrittlement of a 90%Cu-10%Ni alloy and commercially pure nickel.

* The authors are with the Inco Research & Development Center, Inc., Sterling Forest, Suffern, NY 10901.

** The author is with the International Business Machines Corp., Thomas J. Watson Research Center, Yorktown Heights, NY 10598.

High Performance SIMS for
Electronic Material Analysis

C. A. Evans, Jr.
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ABSTRACT

Energetic ion bombardment of the surface of a material causes atoms to be ejected from the near surface region of the material by the well-known sputtering process. A small fraction of these sputtered atoms are ejected as positive or negative ions. Mass spectrometric analysis of these secondary ions provides an analysis of the surface of the sample while continuous monitoring of one or more ions vs. time produces an in-depth profile of the species of interest. Localization of the ion emission can provide microanalysis and the use of the appropriate instrumentation will produce ion images of the lateral distribution of impurities.

Secondary ion mass spectrometry (SIMS) is generally practiced in one of two instrumental modes. The simplest involves attaching a quadrupole mass filter to an UHV system generally in conjunction with another surface analytical technique, both of which use inert gas ion bombardment for sputtering. These SIMS systems provide for surface analysis and well as rudimentary depth profiling. Detection limits are generally 10 to 100 times better than other surface techniques such as AES or ISS.

The alternative approach to SIMS instrumentation is to employ reactive ion bombardment, O_2^+ or Cs^+ , in order to enhance and maximize secondary ion yields and to use a dedicated secondary ion mass spectrometer to efficiently collect and transmit the secondary ions. In this "High Performance" mode, ppb to ppm detection limits are available in the depth profiling mode and lateral distribution images or maps with micrometer resolutions can be generated.

This paper will describe the analytical conditions and instrumentation necessary to obtain this high performance and illustrate these capabilities with examples from Si and GaAs materials research. These illustrations will include topics such as Cr and O redistribution in GaAs and Si (respectively) and studies of intentional electrical dopants in these matrices.

Thursday, 8:30 a.m.

C-1

35

Quadrupole Mass Spectrometer Based Secondary
Ion Mass Spectrometry for Electronic
Materials Characterization

by

Bradway F. Phillips
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Physical Electronics Division Laboratories
6509 Flying Cloud Drive
Eden Prairie, MN 55344

The ability to obtain both good matrix element quantitation by using AES and ESCA, combined with the much higher sensitivity of SIMS for a number of chemical elements important in very low concentration in electronic materials is extremely important. Being able to combine all of these techniques in one instrument which then alleviates the problem of surface contamination when specimens are transferred from one vacuum system to another is even more important as the layer structures used in electronic devices become thinner and careful studies of interface conditions must be performed. This paper will focus on several pertinent SIMS applications and at the same time note the increased characterization abilities that complement AES and ESCA analysis.

The first study to be discussed will be a review of the development of GaAs materials deposition using molecular beam epitaxy and the vital part SIMS analysis played in identifying contaminants and possible sources of contamination as the purity of the GaAs films is improved to device quality.

Secondly, the analysis of thin oxide films and metal silicon interfaces will be discussed. Problems with specimen charging in continuous sputtering situations and AES-SIMS complementary profiling will be discussed.

Third, the analysis of very low energy ion implants for VLSI development will be shown. This area of analysis is extremely important as integrated circuit device density and speed are raised because these two constraints lead to the need for a very near surface profiling technique in which the recontamination rate from the analysis chamber vacuum can lead to erroneous results.

Thursday, 9:20 a.m.

C-2

SIMS ANALYSIS OF LOW TEMPERATURE OHMIC CONTACTS TO GaAs

W. Katz, O. Aina, B. J. Baliga
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and

K. Rose
Rensselaer Polytechnic Institute
Troy, New York

ABSTRACT

The ability to make good ohmic contacts to GaAs is a significant factor in obtaining reliable microwave devices.¹ Consequently, considerable effort has been expended in attempting to make good contacts to GaAs. Most of these involve the alloying of a metal-dopant alloy (i.e. AuGe) with a GaAs substrate at relatively high temperatures (450°C). Such attempts have often resulted in devices displaying unreliable electrical characteristics and/or poor surface morphology.²⁻⁴ The good ohmic contacts that have been fabricated require either the use of molecular beam epitaxy (MBE) to grow a heavily-doped Ge layer⁵ or rather complicated processing (i.e. the use of multilayer encapsulants to minimize Ga and As outdiffusion at the alloying temperature).⁶

We report here a new method for making ohmic contacts to GaAs by a simple low temperature (315-330°C) sintering of AuGe films. Using a four probe method, the specific contact resistivities were measured indicating good contact to GaAs after approximately 3 hours of sintering. SIMS profiles of Au, Ge, Ga, and As were obtained using a Cameca IMS 3-f ion microscope in order to correlate the electrical characteristics with the diffusion profiles. SIMS data indicate significant redistribution of Au and Ge into the GaAs with little Ga and As outdiffusion. Conversely, SIMS analysis of samples alloyed at higher temperatures (approximately 390°C) show considerable outdiffusion of both Ga and As. From the SIMS data the diffusion coefficients of Au and Ge in GaAs were estimated indicating an enhanced diffusion of Ge was responsible for the improved electrical properties. A possible mechanism accounting for the noted enhanced diffusion will be described.

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4. G. Y. Robinson, Solid-St. Electron., 18, 331 (1975).
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6. F. Vidimari, Electronics Lett., 15, 674 (1979).

Thursday, 9:40 a.m.

C-3

37

TANTALUM SILICIDE INTERCONNECT CHARACTERIZATION
BY SURFACE ANALYTICAL TECHNIQUES*

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714 759-2472

ABSTRACT

To reduce the parasitic effects associated with long interconnect lines needed in the present-day VLSI circuits, we have developed a Tantalum Silicide interconnect layer.¹ The physical and electrical properties of these layers were examined using Auger Electron Spectroscopy (AES), Secondary Ion Mass Spectroscopy (SIMS), Scanning Electron Microscopy (SEM) and resistivity and electrical CMOS device parameter measurements. The impact of substrates, annealing conditions, TaSi₂ deposition conditions and pre-deposition cleaning procedures on both the composition of the film and its electrical properties are reported. Using a co-sputtering process, a stoichiometric ratio of Tantalum to Silicon atoms of approximately 1:2 measured using AES is achieved which provides a resistivity of ≤ 5 ohms per square for device and circuit applications. Boron and Phosphorus Ion-implant profiles through TaSi₂ films are examined using SIMS technique to provide the ion-range data.² SEM photographs of surface show good step coverage properties for minimum dimensions near 1 μ m. Results of both physical and electrical parameter evaluations are reported.

* This work was supported by Hughes IR&D program, 1980-81.

1. R. K. Pancholy, K. Y. Chang and C. S. Chen, to be submitted for publication elsewhere.
2. R. K. Pancholy & K. Y. Chang, to be submitted for publication elsewhere.

TiN-Cu Metallization for Solar Concentrator Cells*

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ABSTRACT

The metallization system on Si solar cells used in some satellites consists of a Ti adhesion layer (150 nm), a Pd diffusion barrier (150 nm), and a Ag conductor layer (6 μm). When heated to 600°C for 5 minutes, this metallization interdiffuses with the cell, which decreases the cell output power by 50%. This metallization is not suitable for Si solar concentrator cells used in terrestrial power plants because in these cells the temperature reaches 600°C for 10 minutes during manufacture and 100°C for months during power plant service. Using Auger electron spectroscopy, Rutherford backscattering spectroscopy and transmission electron microscopy with electron diffraction, we have shown that a proposed metallization system consisting of a TiN adhesion-barrier layer (150 nm) and a Cu conductor layer can withstand the 600°C, ten minute exposure without electrical degradation of the cell. From 600° to 700°C Cu diffuses through the polycrystalline TiN layer (average grain diameter ≈ 30 nm) by grain-boundary and other internal surface mechanisms with an activation energy of $Q \approx 400 \text{ kJ} \cdot \text{mol}^{-1}$. This metallization is being tested on commercial Si cells at solar concentrations of 30x and 100x.

*This work supported by the United States Department of Energy (DOE) under contract DE-AC04-76-DP00789.

**A DOE Facility.

Thursday, 10:50 a.m.

C-5

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Spatially Resolved Chemical Analysis
by Auger Spectroscopy

by

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and

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The identification of spatially resolved chemical surface structures using a high resolution Scanning Auger Microprobe (SAM) will be discussed for samples of metallic silicon in a silicon oxide matrix. The spatial imaging of these chemical features is based upon the synergistic use of elemental Auger maps and point source analyses. From the point source analyses, elemental composition can be assigned using both $dN(E)/dE$ and $N(E)$ data. The chemical composition can be confirmed through the use of peak shape and energy analysis of the $N(E)$ data. Quantitative spatial analysis of the silicon oxide/silicon metal surface composition is supported by curve fitting of $N(E)$ data. Analysis conditions for electron beam mapping of silicon oxide independent of artifact formation are constrained by the measured rate of electron beam induced oxide reduction.

Thursday, 11:10 a.m.

C-6

40

IMPROVING THE ACCURACY OF DETERMINATION OF LINE ENERGIES BY ESCA:
CHEMICAL STATE PLOTS FOR SILICON-ALUMINUM COMPOUNDS

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Compounds containing silicon-oxygen and aluminum-oxygen bonds exhibit photoelectron and Auger line energies from the silicon and aluminum that range over about two eV. It is of interest to improve the accuracy of determination of the line energies so that analytically-useful two-dimensional chemical state plots can be constructed.

The kinetic energies of the photoelectrons (from Mg K_{α} photons) and KLL Auger electrons (from the bremsstrahlung) range from 1150 eV to 1617 eV. The Cls line was used as a charge reference at 969 eV (284.6 eV binding energy). In the data acquisition the Cls, Si2p, and Al2p lines were recorded at the start and at the end to compensate for any charging drift, which ordinarily amounted to less than 0.1 eV. The exact (± 0.01 eV) kinetic energy separation of the sputtered silicon 2p and $KL_{23}L_{23}$ Auger lines (462.5 eV) observed on each day was used to correct for any very slight change in the magnitude of the voltage scale. Data on oxides, minerals, and zeolites were determinable to ± 0.02 eV and reproducible to well within 0.1 eV. Good separations of most species on the aluminum and silicon chemical state plots were achievable.

Thursday, 11:30 a.m.

C-7

41

SURFACE ANALYSIS BY FAST ATOM BOMBARDMENT

MASS SPECTROMETRY

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Fast atom bombardment mass spectrometry (FABMS) has been developed in this laboratory to allow routine surface analysis of insulator materials, without the need to resort to the electron beams, which are necessary to neutralise charging effects when analysis is attempted using conventional SIMS. Adverse electron beam effects such as electron stimulated desorption or decomposition, and instabilities due to imperfect charge neutralisation are avoided when atom bombardment is used. FABMS has allowed reliable and reproducible mass spectral analysis of a wide range of insulator surfaces; for example involatile organic compounds and inorganic glasses.

This paper outlines the essential features of the technique and describes a study of the surface and bulk concentrations of the elements Li, B, Ti, and Zr in a series of multicomponent glasses. The data demonstrates that the relative concentrations of these elements with respect to Si can be reliably obtained from the elemental secondary ion intensity ratios M^+/Si^+ .

The studies also show that composition depth profiles vary considerably amongst these elements; for example it is clear that B is depleted in the surface layer whereas there is enrichment in the immediate sub-surface region. Li, however, tends to concentrate at the surface, indeed it is observed to diffuse rapidly to the surface even at room temperature.

Thursday, 11:50 a.m.

C-8

42

A Unified Model for Formation of Schottky Barrier and Oxide-Semiconductor States on 3-5 Compounds and Implications Theory+

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The interface between thin films of metals or oxides on semiconductors is becoming increasingly critical to the operation of semiconductor devices including integrated circuits. The object of the work reported here has been to develop an understanding, on an atomic basis, of the interactions between semiconductor and thin film which determine the electronic characteristics of the interface, e.g. Schottky barrier height or number and energy of oxide-semiconductor interface states.

The principal experimental tool is photoemission excited by monochromatized synchrotron radiation ($10 < h\nu < 300 \text{ eV}$). Extreme surface sensitivity is obtained by tuning the synchrotron radiation so that the minimum escape depth is obtained for the excited electrons of interest. In this way only the last two or three atomic layers of the solid is sampled. By changing $h\nu$, core levels or the valence bands can be studied. Using a metallic reference, the fermi level position at the surface, E_{fs} , can be directly determined.

GaAs, InP, and GaSb have been studied in this work. On a proper cleaved surface, there are no surface states in the semiconductor band gap; thus, no pinning of E_{fs} . Pinning of E_{fs} can then be monitored as metals or oxygen are added to the surface--starting from sub-monolayer quantities. Two striking results are obtained: 1) pinning position is independent of adatom for oxygen and a wide range of metals, and 2) the pinning is completed by much less than a monolayer of adatoms. These results can not rationally be explained by the pinning being due to the levels produced directly by the adatoms. Rather, they suggest strongly that the adatoms disturb the semiconductor surface forming defect levels. This is supported by appearance of the semiconductor atoms in the metal and by the disordering of the semiconductor surface by sub-monolayer quantities of oxygen.

When metals or oxygen are added under very gently conditions, the following levels are formed (all energied relative

III-V	Acceptor(Missing Atom)	Donor(Missing Atom)
GaAs	0.65 eV (As)	0.85 eV (Ga)
InP	0.45 eV (P)	0.1 eV (In)
GaSb	0.5 eV (Sb)	below VBM (Ga)

to the conduction band minimum). These results explain why Schottky barrier gates will provide useful FET's on n-GaAs but not n-InP. Likewise they predict that MOS or MIS gates will be practical for n-InP but not n or p GaAs. Studies of the oxygen surface chemistry find the As oxides to be unstable and P oxides to be stable--reinforcing the prediction. Assuming that similar defects surround dislocations, the low operational life time of typical GaAlAs as compared to GaInAsP lasers can be explained.

+ Work Supported by DARPA and ONR

* Stanford Ascherman Professor of Engineering

Thursday, 1:30 p.m.

D-1

Valence Band Synchrotron Radiation Photoemission
for Ge (111) /"d" metal interfaces: the Ge/Pd,
Ge/Ni, Ge/Ag cases.

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We report the first photoemission studies of the electronic structure of the interface between Ge (111) and "d" metals for monolayer coverages. Three different systems were studied: Ge/Pd, Ge/Ni, and Ge/Ag. In the case of Ge (111) + Pd a broad, reacted, germanide-like phase is formed with similar features to the Si (111) /Pd interface (*). In the Ge (111) + Ni case an evolution of the valence band vs. coverage from a reacted situation to a metal rich situation is found, indicating a strong gradient of concentration; the interface is narrower than in the Ge/Pd case. The third case is the Ge (111) + Ag interface where the intermixing takes place only in a few layers, and a bulk-like Ag growth is quickly gained onto the interface which is very narrow, but not atomically abrupt. Photoemission spectra of the valence band at $h\nu=80$ eV are presented as well as the intensity profiles for the Ge 3d core photoemission versus the metal coverage.

The discussion is carried out on the different physical-chemistry seen in the growth of these three interfaces, indicating analogies and differences to the correspondent silicide-like systems.

(*) I.Abbati, G.Rossi, I.Lindau, and W.E.Spicer in the proceedings of the PCSI 8 conference, 1981

Work supported by the GNSM of CNR, Italy, and by ARPA of DOD (monitored by ONR)

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Thursday, 2:40 p.m.

D-3

44

Studies of implanted GaAs using Raman scattering

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Abstract

Raman spectra were recorded in backscattering from (100) oriented, Cr-doped, semi-insulating crystalline wafers of GaAs. The studies were carried out at room and cryogenic temperatures. Implant fluences up to 1×10^{15} ions/cm² at 120 keV of S and Se were used. Annealing studies were also carried out on the S-implanted wafers. The encapsulating was by means of rf-plasma-deposited Si₃N₄ films. Unprocessed and laser-annealed samples were also studied. Transmission electron micrograph/diffraction data were obtained on selected samples. A pulsed, doubled YAG laser at 532 nm was used with about 16 mW of incident power in a 0.12-mm-diameter spot. A "triple" spectrometer, gated photon counting and computer processing of data were used. The results include the dependence of the LO mode intensity and frequency shift on fluence and annealing, identification of polycrystalline and amorphous regions in the implanted layer, and removal of amorphous regions by laser annealing.

Thursday, 3:30 p.m.

D-4

Effects of Implantation and Annealing on the
Raman Spectrum of InP and GaAs*

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and

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The Raman spectrum of GaAs and InP consists of two peaks corresponding to the longitudinal optical (LO) phonon and the transverse optical (TO) phonon associated with the center of the Brillouin zone. The symmetry of the crystal determines the momentum selection rules. Lattice changes, e.g., ion implantation induced damage, encapsulation and annealing associated strains, and polycrystallite formation, can result in significant differences in the Raman spectrum. We have studied the laser-induced Raman scattering from InP and GaAs as a function of implant fluence and annealing. The Raman spectra of these materials were obtained using a backscattering configuration with a double grating monochromator and the 4880Å and 5145Å lines of an Ar⁺ laser were used for the excitations.

Semi-insulating (100) substrates of InP and GaAs were implanted with Si ions with fluences ranging from 5×10^{12} to $5 \times 10^{15} \text{cm}^{-2}$. Substrates were encapsulated prior to annealing, which was performed at temperatures from 700 to 800°C. The intensity of the LO mode decreased and the line width increased with increasing fluence. Upon annealing an increase in the LO intensity was observed, indicating that the lattice was reordering. In conjunction with the LO mode intensity variations as a function of implant and annealing was the appearance of a well defined signal at the TO frequency. The TO mode is forbidden in the orientation used in this InP and GaAs study and we can only speculate at present on the various possible mechanisms that may be contributing to its existence. Disorder induced Raman spectra of Be in InP and GaAs have also been studied for various fluences and the features observed in the low frequency region have been interpreted on the basis of short range order and phonon density of states. Presented in this paper are the experimental results and discussions on laser-induced Raman scattering from implanted InP and GaAs.

* Work supported by the Naval Research Laboratory Electronics Materials Program and the Office of Naval Research.

Thursday, 3:50 p.m.

D-5

46

MEASUREMENTS OF DEFECTS AND STRAIN IN SOS FILMS
AFTER CW Ar LASER ANNEALING IN THE LIQUID PHASE REGIME

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ABSTRACT

Epitaxial silicon-on-sapphire (SOS) films grown by chemical vapor deposition (CVD) contain a high density of stacking faults and twins, especially near the sapphire interface. Such films are also compressively stressed in the (100) plane. We have melted and epitaxially regrown such films in the msec time regime by means of cw Ar laser irradiation. The regrown films were analyzed by means of optical and secondary electron microscopy, MeV He⁺ Rutherford backscattering and channeling, transmission electron microscopy (TEM), X-ray diffraction, secondary ion mass spectrometry, and surface electrical measurements. These combined techniques have yielded information on surface morphology, defect types and profiles, strain, impurity profiles, and electrical activity. As reported in a previous brief communication¹, MeV He⁺ channeling measurements have shown that the laser annealed Si films, 0.2-0.5 μ m in thickness, had much lower defect densities compared to CVD SOS throughout their thickness. We will present new TEM data which corroborate the channeling results and show that some areas are almost completely defect-free. In addition, X-ray measurements indicate that the room temperature strain in these laser annealed Si films is about ten times lower than in CVD films. The laser annealed films do, however, show some lateral nonuniformity and a p-type doping, which is correlated with an incursion of Al from the underlying sapphire substrate. Detailed results using the above characterization techniques will be presented, and the film growth mechanism will be discussed.

1. I. Golecki, G. Kinoshita, A. Gat, B. M. Paine, Appl. Phys. Lett. **37**, 919 (1980).

* Also Visiting Associate, California Institute of Technology, Mail Code 116-81, Pasadena CA 91125.

Thursday, 4:10 p.m.

D-6 47

Ubiquitous Sodium: Where Does It Come From?

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ABSTRACT

Sodium and other alkali elements appear with great regularity on almost all solid surfaces. Such contamination is viewed with considerable concern since these elements affect many processes or final finished products. In this work the sources of alkali contamination are traced. The sensitive complementary ISS/SIMS methods are used to analyze solid surfaces following various processing steps. Alkali elements, usually sodium occur in the original starting materials or are added in processing, and then concentrate on the surface following grain boundary diffusion. Even when sodium is effectively removed in processing, it is often returned to the surface in the final rinse of so called "high purity" organic solvents. The presence of sodium at surfaces or interfaces in technologies such as adhesive bonding are discussed.

Thursday, 4:30 p.m.

D-7

48

Surface-to-Bulk Optical Absorption Measurements of Laser Windows Using Photoacoustic Chopping Frequency Studies

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Knowledge of the ratio of the surface optical absorption to the bulk optical absorption of a material is desirable for the design of lower absorbing laser windows. If the bulk absorption dominates then the emphasis will be on improving purification of starting materials, crystal growth techniques, using different strengthening additives and on forging methods. If the surface is the major contributor, then the emphasis will be placed on improving polishing and cleaning techniques or on applying protective coatings.

Photoacoustic (PA) chopping frequency studies provide one technique for ascertaining this ratio. There are a number of theories⁽¹⁻³⁾ explaining the PA signal. Experimental chopping frequency variations in the PA signal amplitude and phase have been recorded on a variety of uncoated materials at various wavelengths. From the analysis of the data, information on whether the signal is the result of bulk or surface optical absorption can be obtained. Results will be presented on ZnSe windows at 10.6 μm , and on Al_2O_3 (sapphire) and SiO_2 (fused silica) at 1.3 μm . Calculations were made using the three theories and relevant thermal parameters. In general only the Bennett-Forman theory⁽³⁾ could explain the experimental results.

- (1) A. Rosencwaig and A. Gersho, J. Appl. Phys. 47, 64 (1976).
- (2) F.A. McDonald and G.C. Wetzel, Jr., J. Appl. Phys. 49, 2313 (1978).
- (3) H.S. Bennett and R.A. Forman, J. Appl. Phys. 48, 1432 (1977).

*Work supported by the Materials Laboratory of the Air Force Wright Aeronautical Laboratories, WPAFB, OH 45433.

Thursday, 4:50 p.m.

D-8

On the nature of the redistribution
of Be in annealed GaAs

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We have studied by secondary ion mass spectrometry the redistribution of Be implanted in GaAs at 300 keV and fluences of 4×10^{14} and $4 \times 10^{16} \text{ cm}^{-2}$. We have measured the depth distributions of Be as the GaAs was annealed under a 0.20 μm thick SiO_2 cap for 20 min at 700, 750, 800, 840 and 900°C and 840°C for 5, 10, 20, 40 and 80 min.

For the $4 \times 10^{14} \text{ cm}^{-2}$ fluence, the peak in the Be distribution broadens to a flat distribution with a sharply decreasing deep side distribution, approaching a nearly rectangular doping profile. This effect ceases when 800°C is reached (for 20 min annealing time) or when 20 min is reached (for 840°C annealing temperature). Redistribution then ceases. The equilibrium Be density is between 2 and $3 \times 10^{18} \text{ cm}^{-3}$. The saturation depth for a $4 \times 10^{14} \text{ cm}^{-2}$ fluence at 300 keV is 1.8 μm .

For the $4 \times 10^{16} \text{ cm}^{-2}$ Be fluence, the Be distribution collapses approximately two orders of magnitude directly beneath the peak in the damage depth distribution and Be is retained just above 10^{20} cm^{-3} in the two adjacent regions. The Be redistributes to deeper depths at densities below 10^{19} cm^{-3} , much like for the $4 \times 10^{14} \text{ cm}^{-2}$ fluence. However, it does not saturate with either time (at 840°C) or with temperature, but the Be migration does not proceed with time according to the expression $x^2 = (Dt)^2$ nor with temperature according to $x^2 = x_0^2 \exp(-\frac{E_a}{kT})$ as it would for normal diffusion.

We conclude that for a fluence below the amorphization fluence for Be in GaAs, for example $4 \times 10^{14} \text{ cm}^{-2}$ at 300 keV at room temperature, Be migrates (redistributes) with time or temperature by a density-driven mechanism toward a saturation density of about $2 \times 10^{18} \text{ cm}^{-3}$, and to whatever depth is determined by the total Be fluence implanted and no further redistribution occurs with time or with temperature, up to at least 900°C. For a fluence above the amorphization fluence, for example $4 \times 10^{16} \text{ cm}^{-2}$ for 300 keV Be at room temperature, the Be density is reduced in the region of GaAs lattice regrowth upon annealing (thermal processing), to a saturation density of $< 10^{19} \text{ cm}^{-3}$ in undamaged GaAs.

The migration of Be during this process is not be standard diffusion and cannot be characterized by a diffusion coefficient. It appears to occur by a density-driven mechanism, rejection from the region where the density exceeds the saturation value, and movement into a region where the density becomes the saturation value and the depth is the fluence divided by the saturation density, $\sim 2 \times 10^{18} \text{ cm}^{-3}$ in the case studied here in detail. The nature of this Be redistribution in GaAs is similar to that of Mg in GaAs.

Thursday. 5:10 p.m.

The Use of Analytical Surface Tools in the Fundamental Study
of Adhesion, Friction and Wear

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ABSTRACT

The real area of contact between two solid surfaces is only a small portion of the apparent area. Deformation of these areas can result in solid state contact through surface films. For clean solid-to-solid contact strong adhesive bonding occurs across the interface. Under these conditions many properties of the solid such as the metallurgical and chemical nature of metals can influence adhesion, friction and wear behavior. The presence of gases, liquids and solid films on the surface of solids markedly alters the tribological characteristics. These surface films can also considerably change the mechanical effects of solid state contact on bulk material behavior.

This paper reviews the various techniques and surface tools available for the study of the atomic nature of the adhesion, friction and wear of materials. These include chemical etching, X-ray diffraction, electron diffraction, scanning electron microscopy, low-energy electron diffraction, Auger emission spectroscopy analysis, electron spectroscopy for chemical analysis, field ion microscopy, and the atom probe. Properties of the surface and wear surface regions which effect wear such as surface energy, crystal structure, crystallographic orientation, mode of dislocation behavior, and cohesive binding are discussed. A number of mechanisms involved in the generation of wear particles are identified with the aid of the aforementioned tools.

Friday, 8:30 a.m.

E-1

51

AUGER COMPOSITIONAL ANALYSIS OF GEAR STEEL REACTED WITH TRICRESYL PHOSPHATE

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Previous studies by Auger electron spectroscopy (AES) have established that a correlation exists between improved ball-bearing performance and the surface compositional changes induced in the bearing steels by soaking in warm, 100% tricresyl phosphate (TCP). Because the use of a TCP presoak can eliminate the need for assembling components to undergo mechanical "run-in," the treatment is promising for application by the manufacturer to separate components destined for field assembly or replacement.

The TCP treatment has been proposed for other types of steel mechanisms. Before any mechanical evaluation is undertaken, however, analysis by AES is recommended to determine whether the TCP treatment does induce surface compositional changes comparable to those deemed responsible for the performance improvement of the high-carbon, ball bearing steels. For TCP-treated ball bearings, the significant extension of low-speed lifetime under boundary (i.e., thin film) lubrication conditions was ascribed to the introduction of a limited amount of phosphorus-oxygen moiety in the outermost oxide layer of the steel. With increased time of exposure to TCP, depth of penetration into the oxide (rather than concentration) of the P-O moiety increased.

The current study was undertaken to determine the response to TCP treatment of a case-hardened, low-carbon gear steel. Comparison of AES results on case-hardened AISI/SAE 9310 steel Ryder gears with and without a 15-day immersion in 100% TCP at 110°C showed the treatment to be successful in introducing a few atom percent of phosphorus in the outer, predominantly iron-oxygen surface layer. The lineshape of the low-energy phosphorus Auger feature was consistent with that of a P-O moiety. Unlike the results on the as-received 9310 steel, where no P was detected in the oxide layer, depth profiling of the TCP-soaked steel indicated persistence of the P-O moiety throughout the oxide layer, with the P concentration still greater than one atom percent at the oxide/metal interface. These observations compare favorably with the AES results obtained on 52100 ball bearing steels under TCP treatment conditions leading to optimum bearing performance. It remains for performance evaluation tests to establish whether the compositional changes in 9310 gear steel translate into improvement in operational lifetimes for spur gear sets under load.

Friday, 9:20 a.m.

E-2

52

ABSTRACT

Surface Studies of Corrosion Preventing
Coatings for Aluminum Alloys

by

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Corrosion inhibiting coatings (i.e., pretreatments) are used in the production of painted aluminum surfaces. This paper compares the surface compositional changes as they occur during the chemical coating of 3003 aluminum alloy. Several commercial coatings are examined and chemically identified by a combination of Auger electron spectroscopy, scanning electron spectroscopy, Fourier transform infrared spectroscopy and X-ray energy dispersive spectroscopy.

Corrosion inhibiting coatings are classified based on their chemical composition. Commercial pretreatments include at least two types: a) those which chemically react with the alloy surface and produce a chemically different protective layer without regard to effluents, and b) coatings designed to decrease environmental problems (i.e., cyanide-free), while still producing a protective layer.

Friday, 9:40 a.m.

E-3

53

CORROSION PROTECTION OF ALUMINUM BY SOLUTION-DEPOSITED MIXED OXIDE FILMS

by

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We have made an extensive study of the use of solution-deposited "mixed" oxide coatings to enhance the corrosion resistance of aluminum and aluminum alloys. A "mixed oxide" is defined for this purpose as an oxide system containing more than one cation species. The solution-deposited mixed oxides studied are broadly categorized according to whether the valence of the depositing cation does or does not undergo a formal change during film formation. We discuss film deposition and formation procedures from both aqueous and non-aqueous solutions of various metal salts and metal organic compounds.

Deposited coatings were characterized using Auger electron spectroscopy and x-ray photoelectron spectroscopy. Corrosion resistance of coatings was evaluated through exposure in a salt spray chamber.

The general suitability of new types of mixed oxide systems for the corrosion protection of aluminum is discussed. Baseline data obtained on widely used chromate coating formulations are used for comparison. The question of the uniqueness of our interpretation of experimental Auger depth profiles in terms of the existence of a mixed oxide layer is also addressed.

Friday, 10:00 a.m.

E-4

54

ADHESIVE BOND DURABILITY WITH CONVERSION COATINGS

by

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ABSTRACT

The effect of processing time during conversion coating of aluminum alloys on adhesive bond durability was investigated. Iridite 14-2 process, both by immersion and brush application methods, and Alodine 1500 processes were studied and compared with "optimized" Forest Product Laboratory (FPL) process of surface preparation. The surface morphology and thickness of conversion coatings were studied by Scanning Transmission Electron Microscopy (STEM). The chemical composition of the surfaces were determined by Electron Spectroscopy for Chemical Analysis (ESCA) in an Auger Electron Spectrometer (AES). Bond durability was determined by performing wedge tests. It was found that the crack extensions for leached Iridite 14-2 and brush Iridite 14-2 coatings were comparable with those of "optimized" FPL surfaces. However for Alodine 1500 and non-leached Iridite 14-2 methods of surface preparation, crack extensions were much greater than leached Iridite and brush Iridite processes which seem to suggest that with some sacrifice in corrosion protection better adhesive bond durability could be achieved. The durability results were correlated with the morphology and the chemical composition of the conversion coatings.

Friday, 10:50 a.m.

E-5
55

APPLICATIONS OF SOFT X-RAYS IN SURFACE ANALYSIS

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Capabilities of Low-Energy Electron-Induced X-ray Spectroscopy (LEEIXS) for providing detailed qualitative and quantitative informations about the first 10 to 1000 Å of a solid surface are emphasized. The observation of characteristic soft X-rays produced by the bombardment of materials with low-energy electrons (1-5 keV) forms the basis of the LEEIXS system in which the central feature is a cold cathode tube associated with a nearly classical wavelength dispersive X-ray spectrometer. As shown by the following examples the LEEIXS technique can be applied for studying a wide range of substantially different analysis problems.

Trace detection of low Z elements such as B and F in thin films formed on titanium substrates is accomplished. These impurities are incorporated from the electrolytes (H_3BO_3 and H_2SiF_6 solutions respectively) during anodization. Such investigations are necessary in the aircraft industry in order to establish more reliable bases on metals or alloys for adhesive bondings. For the same purpose, characterization of thin films formed on titanium by chemical conversion in phosphate/fluoride solutions is also investigated. Chemical bonding informations are obtained from the fine structure of the OK_{α} and $TiL_{2,3}$ X-ray emission bands. Results prove unambiguously that the main component of the conversion films is not a TiO_2 oxide. In addition LEEIXS has been used for getting O and P elemental depth profiles through an anodic film grown on niobium. Such an investigation has been performed in conjunction with an external step by step chemical etching of the specimen surface.

Lastly it is reported additional LEEIXS applications dealing with the surface characterization of stainless steels which have suffered various passivation treatments and of microelectronics industry devices which have been externally heat treated.

Friday, 11:10 a.m.

E-6

56

The Hydration of Phosphoric Acid Anodized
Aluminum Surfaces:
A Surface Phase Diagram Determined by XPS*

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Abstract

The surface composition of Al oxide formed by phosphoric acid anodization (PAA) during different stages of hydration is determined by x-ray photoelectron spectroscopy (XPS). The evolution of the oxide composition during exposure to water is traced on the P_2O_5 - H_2O - Al_2O_3 ternary phase diagram and is found to proceed in three stages. The first is a precursor step involving the conversion of an initial monolayer of $AlPO_4$ to $AlPO_4 \cdot H_2O$. It can occur readily during storage as a result of incomplete drying of the sample and involves no changes in the oxide morphology. In the second step, a pseudo-boehmite ($AlOOH$) phase nucleates and grows on the surface after a period of incubation; the surface composition follows the tie line connecting the initial point ($AlPO_4 \cdot H_2O$) and $AlOOH$. Scanning transmission electron micrographs also show that the oxide surface becomes covered by a pseudo-boehmite hydration product that increases in thickness with exposure time. The final hydration step occurs as crystallites of bayerite ($Al(OH)_3$) nucleate and grow on top of the pseudo-boehmite; the surface composition follows the Al_2O_3 - H_2O tie line.

The advantages of using XPS to determine the hydration phase diagram are twofold. First, since surface rather than bulk quantities are measured, XPS is very sensitive to small changes occurring during the early stages of surface hydration. Second, by determining the surface chemical composition and its effect on the hydration kinetics, we can predict the effectiveness of a surface treatment in retarding hydration. For this and similar systems we find that the surface phosphate coverage is a major factor in determining the incubation time between the first and second stages. Surfaces of Al oxide with a higher phosphate coverage resist the growth of pseudo-boehmite for longer periods of time.

* Supported by AFOSR under contract No. F-49620-78-C-0097.

⁺ Present address: Alcoa Technical Center, Alcoa Center, PA 15069

ABSTRACT

Thermal Diffusion of Magnesium in Several Aluminum Alloys

by

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Several types of staining are observed on the surfaces of rolled aluminum coils. Two of these, white lacy stain and dirty metal, are caused by a localized increase in formation of a hydrated aluminum oxide and magnesium oxide, respectively. This contribution examines the formation of these oxides on ingots, particularly MgO.

Three aluminum alloys with varying bulk magnesium concentration were examined: 5052, 3004 and 3004 with 2.64, 0.96, and 0.03% magnesium, respectively. Samples were prepared and heat-treated in flowing air at varying temperatures and times. The surfaces were then examined with Auger electron spectroscopy to determine the magnesium and aluminum present in the oxides formed. These results were then correlated with the temperature and time of the heat-treatment as well as the surface morphologies as seen with ultra-high resolution scanning electron microscopy. This information can be used to predict the tendency of an alloy to produce a particular type of staining.

As expected, the alloys with higher bulk magnesium contents (i.e., 5052 and 3004) showed more magnesium diffusion and magnesium oxide formation on the sample surface. However, this same type of diffusion and oxide formation was also seen on the softer 3003 alloy. At high temperatures, the surface of 5052 tends to be almost pure MgO.

Friday, 11:50 a.m.

E-8

58

Abstract

ELECTRON BEAM INDUCED DAMAGE IN ITO COATED KAPTON, I. Krainsky, W. L. Gordon, and R. W. Hoffman, Department of Physics, Case Western Reserve University, Cleveland, Ohio 44106.

We report data for the stability of thin conductive indium tin oxide (ITO) films on 0.003 in. thick Kapton substrates during exposure of the surface to electron beams. 20 nm of ITO was deposited by sputtering with subsequent annealing and supplied by G. E. for NASA. There are several phenomena which can play a role in electron beam effects; particularly electron stimulated desorption (ESD); electron stimulated decomposition and adsorption (ESA); and thermal effects including diffusion and segregation. All of these effects can be the cause of a change of surface properties. Different current densities of the 3 KeV primary electron beam from 2×10^{-6} A/cm⁻² to 7×10^{-3} A/cm⁻² were used. It was found that the larger primary beam intensities can cause a rupture of the ITO films and even melting of the Kapton substrate. The secondary emission coefficient and AES spectra were obtained as a function of time for the different beam intensities. In all cases beam exposure results in a decrease of the secondary yield but because of thermal effects this change, as well as composition changes, cannot be directly interpreted in terms of electron beam dosage. To date the Auger data show an inverse correlation of the SEE with S concentration on the surface, but no dependence on C, O, or other elements even though the absolute quantity of S is small. Light sputtering of the ITO layer shows that the S is a surface contaminant. The stability of the secondary yield is increased after sputtering.

* Supported by NASA Grant NSG 3197.

Friday, 12:10 p.m.

F-9

59

ANALYSIS OF AIRCRAFT FUEL LINE DEPOSITS BY POLARIZATION INFRARED
FOURIER MICRO-EMISSION SPECTROPHOTOMETRY

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Circulating fuel is also used as a heat transfer medium in modern aircraft. An accumulation of even very thin layers of deposits can be very harmful to effective heat exchange. Identification of the nature of deposits and the mechanism by which they are laid down can therefore be of help in an amelioration of this problem.

Conventional infrared emission spectrophotometry of the deposits on their original support is an attractive procedure. It will, however, generally fail because of excessive blackbody radiation; i.e. only a continuum with little super imposed structure is seen in the spectra. Fourier spectrophotometry (Michelson interferometry) is more sensitive, so that the temperature differential between sample and detector can be quite small, but it may still fail because of high blackbody background.

In our procedure a slow-scanning Fourier infrared Michelson interferometer was converted into an emission microspectrophotometer by replacement of the commercial source with a reflecting microscope objective (32X) focussed on an deposits on a metal surface maintained at a temperature somewhat higher than that of the detector and with appropriate optical interfaces. In the normal mode of operation the Golay cell detector was coupled to a chopper consisting of a tuning fork with reflecting tines located in such a way as to furnish a signal representative of the difference between the sample radiation and that of a blackbody reference. Substitution of the chopper by a polarizing disc consisting of closely spaced parallel wires, which was rotated about the optic axis between sample and lens, gave a signal proportional to the difference between sample radiation polarized parallel and perpendicular to the plane of incidence of the beamsplitter. Graybody radiation of any origin was thus effectively subtracted out, with the result of making the instrumentation considerably more sensitive to polarizable infrared emission bands. Furthermore, rotation of the deposits with respect to the beamsplitter's plane of incidence would usually reveal a preferred orientation at a definite "phase angle". This orientation could be correlated to a temperature gradient existing on the substrate at the time of deposition.

Rather remarkable spectral contrast could be achieved because (i) the objective looked at individual grains or clusters of grains and avoided blackbody radiation from the interstices and (ii) the polarization method would discriminate against random blackbody radiation altogether. However, since the polarization spectra are differences, their interpretation requires much care and series of spectra must be compared.

This work was supported by the Air Force Office of Scientific Research under Grant No. 78-3473-12-81.

Friday, 1:30 p.m.

F-1

A Versatile Sample Isolation, Chemical Modification and
Introduction System Designed For A Physical Electronics
Model 548 Electron Spectrometer

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X-ray Photoelectron spectroscopy (XPS or ESCA) has been extensively used in recent years to investigate the chemical nature of various surfaces. In our laboratory it has been used primarily to monitor elemental atomic ratios and binding energy shifts during various modifications of electrode surfaces introduced through gas phase radio frequency plasma or chemical reactions. To maintain surface integrity for XPS analysis by avoiding exposure of the sample surface to oxygen or contaminants in the ambient room atmosphere, a versatile sample treatment and transfer system has been built and attached to the central chamber of a Physical Electronics Model 548 electron spectrometer. This system is presently much more suited to our needs than any commercial or previously reported unit. The system consists of two sample transfer systems with magnetically coupled transfer rods which have sample carriers optimized for right angle transfers. The chemical modification section consists of a differentially pumped radio-frequency plasma chamber, a sample introduction lock, and a chemical treatment chamber which can be used for electrochemical measurements. Each of the chambers of the sample treatment section and the two transfer systems can be isolated using ultra-high vacuum gate valves. The sample mounting, transfer and transport mechanisms, and system performance will be discussed. Examples of XPS analysis of glassy carbon surfaces treated using RF plasmas will be presented for illustrative purposes.

Friday, 2:10 p.m.

F-3

61

Abstract

SURFACE VISCOUS FLATTENING KINETICS OF AMORPHOUS SELENIUM BY OPTICAL SCATTERING TECHNIQUE. S. E. Hebboul, R. W. Hoffman, Department of Physics, Case Western Reserve University, Cleveland, Ohio 44106, and Frank Jansen, Xerox Corporation, Webster, New York, 14580.

The surface flattening kinetics of an amorphous Se film surface exhibiting a periodic grating structure are investigated ex-situ using a simple new optical scattering set-up. The pp and ss scattered light distributions obtained at normal incidence were alternately recorded by means of a continuously rotating pair of optical fiber cables while the Se sample surface temperature was held constant at 42°C. The scattering probability per unit solid angle varied from 10^{-2} to 10^{-6} and was measured by a source-matched photomultiplier tube. Typically, one pair of successive pp and ss distribution curves was obtained in a time span of 130 sec. Therefore, it was possible to record a detailed history of the dynamic changes that occurred in the impressed surface profile of the Se sample during extended periods of time. Initially, higher order diffraction peaks were present for both the pp and ss polarization modes indicating a non-sinusoidal surface profile. However, their peak heights became rapidly negligible compared to the two main diffraction peaks which are characteristic of a sinusoidal surface pattern. Assuming the validity of first order (FO) scattering theory, the decay of the remaining diffraction peak heights at 42°C revealed that the amplitude of the resulting sinusoidal profile decayed exponentially with time while its wavelength stayed constant. Using the results of Mullins¹ calculations and assuming viscous flow to be the dominant transport process, a value of 4×10^{-8} cm. sec⁻¹ was obtained for the ratio of the surface tension γ to the viscosity η at 42°C. One advantage of this technique is that by deducing the decay rate from the higher order diffraction peaks, the assertion that the flattening is controlled by viscous flow is easily tested since different processes have different grating spacing dependences. Also, the applicability of FO theory is easily verified by measuring the ratio of the pp to ss scattering probabilities and comparing with theory. Work is underway to determine the temperature dependence of the ratio γ/η for Se.

¹W. W. Mullins, J. Appl. Phys. 30, 77 (1959).

CITRIC ACID AUGMENTED FLASHLAMP CLEANING
OF
CORRODED STEEL SURFACES

by

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Abstract

Due in large part to its environmental acceptability, solutions of citric acid are used to effectively attack and dissolve rust while maintaining the integrity of the base metal. However, the need for high temperatures and solution additives poses constraints. We have recently demonstrated an improved method of rust removal that synergistically employs the high intensity light output of a flashlamp together with a thin layer of citric acid solution. Such a combination will remove rust from steel surfaces as effectively as conventional citric acid cleaning, but at an increased rate. The short, intense burst of radiation in conjunction with the chelating properties of the citric acid acts to explosively remove surface oxide layers. Microscopic surface analysis (scanning electron microscopy and X-ray diffractometry) reveals conversion of red hematite to black magnetite during the irradiation process. Subsequent applications of the treatment serves to completely remove the magnetite leaving a clean metal surface. Optical radiation cleaning of steel surfaces offers several advantages: ambient temperature citric acid solutions are used in small quantities, organic surface contaminants are removed in the process, and an increased resistance of the cleaned surface towards flash rusting. Details of the flash cleaning process and examples of treated surfaces will be presented in this paper.

Friday, 2:50 p.m.

F-5

63

THE USE OF BACKSCATTER ELECTRON IMAGING IN INSPECTION AND
EVALUATION OF THICK FILM CIRCUITRY

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ABSTRACT

Scanning Electron Microscopy utilizing Secondary Electron Imaging (SEI) has been found, over the years, to be very useful in inspecting hybrid microelectronics circuitry. A relatively new addition to the field of Scanning Electron Microscopy is Robinson Backscatter Electron Imaging (RBEI). It has been found that RBEI enhances the usefulness of SEM in the evaluation and inspection of various aspects of thick film circuitry. RBEI has the ability to probe more deeply into the specimen than SEI and also discriminates between elements with different Z numbers by a difference in shade. Because of the ability of RBEI to probe more deeply into the specimen, extremely small sub surface or sealed over microcracks were discovered during an investigation that was performed on laser trimmed resistors printed on multilayer dielectric. These cracks were undetected using SEI. Another use of RBEI in inspection of multilayer thick film circuitry is made possible because of its ability to discriminate between elements by shade. The circuit can be crosssectioned and the various layers can be distinguished using RBEI. If some element of the circuit, such as a dielectric is made of several distinct compounds which contain elements of different atomic number, it will show up as a "salt and pepper" type mixture. Studies of the area under the conductors of cross sectioned circuits show high z number material existing along the grain boundaries and in the pores of the alumina substrate. It was theorized and demonstrated by X-ray analysis techniques that the material was contributed by the flux portion of the conductor. Samples processed at higher temperatures or for longer periods of time show marked increases in diffusion in depths. In our investigation of phenomenon we found certain conductors had diffusion of material as far as 50 microns into the substrate after 45 minutes at 985°C. Another way that RBEI has been found to be useful in inspection of thick film materials is in distinguishing an impurity imbedded in the surface of a conductor or dielectric. Certain impurities if they do not protrude from the surface are not seen with SEI but appear as lighter or darker areas in RBEI. The impurity can then be identified by the use of X-ray analysis techniques.

Friday, 3:10 p.m.

F-6

64

THE HEIGHT SENSITIVE SURFACE ROUGHNESS PARAMETERS

BY

RICHARD B. ZIPIN

ABSTRACT

Surface roughness can be characterized by several different profile height sensitive parameters. For any particular surface profile waveform all of the height sensitive parameters are simply related and are readily calculated, one from another. Thus, in a production inspection environment wherein the surfaces being compared are all produced by the same process and therefore all have nearly the same waveform shape, any single height sensitive parameter is sufficient for comparison provided there is some knowledge about the shape of the waveform being produced by the manufacturing process. This generally allows the use of averaging rather than profiling instruments.

The definitions of the various parameters of interest are examined and the parameters are calculated for a wide range of idealized profile waveforms, one of which may be chosen to approximate almost any actual surface profile. Using the relationships presented, a measurement of any one of the parameters of interest, together with an assumption about the waveform shape, may be used to determine all of the other desired parameters. The relationships presented are also extremely useful for estimating the various roughness parameters from examination of profile charts.

Friday, 3:30 p.m.

F-7

65

Title: A Study of 946A Connector Thermal-Compression Bond Failure
as Related to Surface Texture

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An Abstract

Thermal-Compression (TC) bonding is a heat-pressure process wherein gold plated components are joined by atomic migratory fusion across their gold interface. The method is widely used for assembling micro size electronic elements.

Properly performed, clean, sound junctions are achieved without the deleterious contamination inherent in methods using flux and other chemicals.

In practice, optimum TC bonds are not always achieved. Historically, the fault is charged to chemical-metallurgical ills: plating bath residues, air-borne substances, handling, etc. However, as described in this paper, surface physical texture is also a direct cause.

At 6,000 diameters, gold plated surfaces exhibit widely different textures depending upon specific plating parameters: Bath chemistry can be adjusted to yield textures (descriptively tagged) from "smooth cobblestone" to "coarse tangled straw." Similarly, plating mechanical-electrical configurations can be adjusted to yield textures from "smooth wet beach sand" to "coarse shot."

A mathematical method of comparatively indexing the different textures is described herein on a scale of 1 to 50: One is extremely fine; 50 is extremely coarse.

This is important because TC bond strength is maximum at Index No. 1 and decreases continuously to minimum (or zero) as conditions approach Index No. 50.

In Summary: Chemical-metallurgical contamination is a cause of TC bond failure. Gold surface texture is another cause. Spectrographic analysis fails to detect the latter cause, and no amount of cleaning can remedy the defect. It can be controlled however by knowledgeable adjustment of plating parameters.

* * * * *

Friday, 3:50 p.m.

F-8

66

RESIDUAL CONTAMINATION AND CORROSION ON
ELECTROCHEMICALLY MARKED URANIUM

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ABSTRACT

Residual contamination and potential corrosion problems on uranium parts resulting from PHB-1 and PHB-1E electroetchants have been investigated using ion microprobe mass analysis (IMMA), scanning electron microscopy (SEM), and light microscopy (LM). The effectiveness of various solvent cleaning sequences and the influence of the use of an abrasive cleaner were evaluated. The marking thicknesses and chlorine distributions were determined.

(a) Operated for the Department of Energy by Union Carbide Corporation-Nuclear Division under Contract W-7405-eng-26.

Friday, 4:10 p.m.

F-9

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SECTION V
SYMPOSIUM CONCLUSIONS

The purpose for holding this Symposium was to bring together basic research, applied research and problem solving efforts all involving some type of surface analysis of Air Force interest into one meeting to try to improve communications and understanding of persons in these various endeavors. That a need for such a meeting exists seems, more and more, to be established from the many favorable comments received and from the growing interest in this Symposium.

The Air Force Office of Scientific Research is to be commended for supporting this Symposium.

